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5-Methoxy-2-[(2-morpholinoethyl)-iminiomethyl]phenolate

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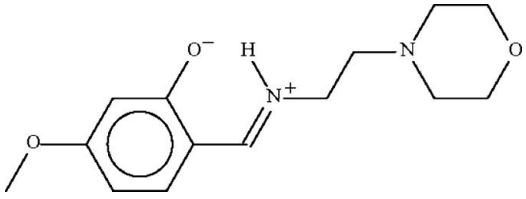
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.122; data-to-parameter ratio = 16.8.

Each of the two independent molecules of the title comound, $C_{14}H_{20}N_2O_3$, exists in the zwitterionic form as the imino N atoms are protonated. The =N—H unit forms an intramolecular hydrogen bond to the negatively charged O atom, and also a weaker intermolecular N—H···O bond, the latter resulting in inversion dimers.

Related literature

For the structure of 2-[(2-morpholinoethylimino)methyl]-phenol, see: Petek *et al.* (2005).



Experimental

Crystal data

$C_{14}H_{20}N_2O_3$
M_r = 264.32
Triclinic, $P\overline{1}$
a = 10.4022 (2) Å
b = 10.7340 (2) Å
c = 14.3497 (3) Å
α = 83.523 (1)°
β = 74.810 (1)°
γ = 60.768 (1)°
V = 1349.13 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 100 K
0.25 × 0.25 × 0.25 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: none
9402 measured reflections
5948 independent reflections
4990 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.122
S = 1.04
5948 reflections
353 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.88 (1)	1.96 (1)	2.6489 (15)	133 (2)
N1—H1···O1^{i}	0.88 (1)	2.32 (1)	2.9570 (18)	129 (1)
N3—H3···O4	0.89 (1)	2.02 (1)	2.6930 (15)	132 (1)
N3—H3···O4^{ii}	0.89 (1)	2.29 (1)	2.9505 (18)	132 (1)

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $-x+3, -y, -z-1$.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

The authors thank the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2422).

References

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supplementary materials

Acta Cryst. (2009). E**65**, o1067 [**doi:10.1107/S1600536809013786**]

5-Methoxy-2-[(2-morpholinoethyl)iminiomethyl]phenolate

N. Mohd Lair, H. Mohd Ali and S. W. Ng

Experimental

2-Hydroxy-4-methoxybenzaldehyde (0.3 g, 2 mmol) and *N*-(2-aminoethyl)morpholine (0.26 g, 2 mmol) were heated in acidified ethanol (50 ml) for 8 h. The solvent was removed to give an oil; crystals appeared in the oil after several days.

Refinement

H atoms were placed at calculated positions (C—H = 0.95–0.99 Å) and were treated as riding on their parent C atoms, with U(H) set to 1.2–1.5 times U_{eq}(C). The iminium H atoms were located in a difference Fourier map, and were refined with a distance restraint of N—H = 0.88 (1) Å; their temperature factors were refined.

Figures

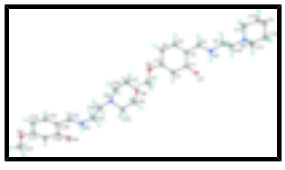


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of the two independent molecules of $C_{14}H_{20}N_2O_3$ at the 70% probability level. H atoms are drawn as spheres of arbitrary radius.

5-Methoxy-2-[(2-morpholinoethyl)iminiomethyl]phenolate

Crystal data

$C_{14}H_{20}N_2O_3$	$Z = 4$
$M_r = 264.32$	$F_{000} = 568$
Triclinic, $P\overline{1}$	$D_x = 1.301$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 10.4022$ (2) Å	Cell parameters from 4712 reflections
$b = 10.7340$ (2) Å	$\theta = 2.3$–$28.3°$
$c = 14.3497$ (3) Å	$\mu = 0.09$ mm^{-1}
$\alpha = 83.523$ (1)°	$T = 100$ K
$\beta = 74.810$ (1)°	Block, yellow
$\gamma = 60.768$ (1)°	0.25 × 0.25 × 0.25 mm
$V = 1349.13$ (5) Å^3	

Data collection

Bruker SMART APEX diffractometer	4990 reflections with $I > 2\sigma(I)$

Radiation source: fine-focus sealed tube	$R_{int} = 0.019$
Monochromator: graphite	$\theta_{max} = 27.5°$
$T = 100$ K	$\theta_{min} = 1.5°$
ω scans	$h = -13 \rightarrow 13$
Absorption correction: none	$k = -13 \rightarrow 13$
9402 measured reflections	$l = -18 \rightarrow 16$
5948 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.040$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.122$	$w = 1/[\sigma^2(F_o^2) + (0.0696P)^2 + 0.3249P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} = 0.001$
5948 reflections	$\Delta\rho_{max} = 0.31$ e Å^{-3}
353 parameters	$\Delta\rho_{min} = -0.28$ e Å^{-3}
2 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.98316 (10)	0.43387 (9)	0.41326 (7)	0.0206 (2)
O2	0.96310 (10)	0.15768 (9)	0.18272 (6)	0.0212 (2)
O3	0.38917 (12)	0.92759 (12)	0.95202 (8)	0.0352 (3)
O4	1.50070 (10)	−0.11554 (10)	−0.55972 (7)	0.0248 (2)
O5	1.47841 (10)	−0.36606 (9)	−0.80663 (6)	0.0207 (2)
O6	0.93660 (11)	0.37780 (10)	−0.02785 (6)	0.0226 (2)
N1	0.77639 (12)	0.49804 (11)	0.57933 (8)	0.0179 (2)
H1	0.8542 (15)	0.5066 (19)	0.5426 (11)	0.039 (5)*
N2	0.60959 (11)	0.75546 (11)	0.78788 (7)	0.0168 (2)
N3	1.23596 (12)	0.09777 (11)	−0.46514 (8)	0.0201 (2)
H3	1.3361 (10)	0.0512 (16)	−0.4734 (12)	0.034 (5)*
N4	1.02064 (11)	0.24956 (10)	−0.21452 (7)	0.0163 (2)
C1	0.91918 (13)	0.37426 (12)	0.38550 (9)	0.0157 (2)
C2	0.98177 (13)	0.29765 (12)	0.29480 (9)	0.0165 (2)
H2	1.0732	0.2909	0.2540	0.020*
C3	0.91073 (14)	0.23419 (12)	0.26664 (9)	0.0165 (2)
C4	0.77322 (14)	0.24178 (12)	0.32475 (9)	0.0178 (3)
H4	0.7249	0.1988	0.3032	0.021*
C5	0.71188 (13)	0.31159 (12)	0.41178 (9)	0.0174 (2)
H5	0.6208	0.3158	0.4512	0.021*
C6	0.78113 (13)	0.37826 (12)	0.44499 (9)	0.0157 (2)

C7	1.11022 (15)	0.12990 (14)	0.12544 (9)	0.0226 (3)
H7A	1.1382	0.0687	0.0699	0.034*
H7B	1.1850	0.0817	0.1647	0.034*
H7C	1.1080	0.2203	0.1026	0.034*
C8	0.72030 (14)	0.43854 (12)	0.53895 (9)	0.0171 (2)
H8	0.6315	0.4357	0.5759	0.021*
C9	0.72088 (15)	0.54438 (13)	0.68036 (9)	0.0193 (3)
H9A	0.8066	0.5011	0.7122	0.023*
H9B	0.6470	0.5115	0.7139	0.023*
C10	0.64529 (14)	0.70646 (13)	0.68841 (9)	0.0180 (3)
H10A	0.7136	0.7401	0.6460	0.022*
H10B	0.5506	0.7489	0.6657	0.022*
C11	0.54085 (16)	0.91155 (14)	0.78853 (10)	0.0248 (3)
H11A	0.4522	0.9524	0.7595	0.030*
H11B	0.6154	0.9399	0.7486	0.030*
C12	0.49055 (17)	0.97165 (15)	0.88980 (10)	0.0297 (3)
H12A	0.5807	0.9394	0.9161	0.036*
H12B	0.4397	1.0772	0.8877	0.036*
C13	0.46395 (18)	0.77580 (18)	0.95435 (10)	0.0333 (4)
H13A	0.3965	0.7439	0.9998	0.040*
H13B	0.5568	0.7415	0.9780	0.040*
C14	0.50611 (16)	0.71168 (16)	0.85489 (10)	0.0271 (3)
H14A	0.5558	0.6062	0.8584	0.032*
H14B	0.4134	0.7446	0.8313	0.032*
C15	1.42646 (14)	−0.13723 (13)	−0.60736 (9)	0.0178 (3)
C16	1.49985 (14)	−0.24612 (12)	−0.68219 (9)	0.0176 (3)
H16	1.6069	−0.3044	−0.6959	0.021*
C17	1.41790 (14)	−0.26689 (12)	−0.73376 (9)	0.0168 (2)
C18	1.25664 (14)	−0.18526 (13)	−0.71453 (9)	0.0187 (3)
H18	1.2014	−0.2031	−0.7497	0.022*
C19	1.18346 (14)	−0.08089 (13)	−0.64448 (9)	0.0184 (3)
H19	1.0762	−0.0254	−0.6318	0.022*
C20	1.26264 (14)	−0.05270 (12)	−0.59010 (9)	0.0172 (2)
C21	1.64017 (15)	−0.44652 (14)	−0.83657 (10)	0.0248 (3)
H21A	1.6685	−0.5143	−0.8881	0.037*
H21B	1.6807	−0.4988	−0.7815	0.037*
H21C	1.6821	−0.3815	−0.8606	0.037*
C22	1.17933 (14)	0.05782 (13)	−0.51997 (9)	0.0185 (3)
H22	1.0723	0.1080	−0.5116	0.022*
C23	1.14126 (15)	0.21047 (13)	−0.39115 (9)	0.0216 (3)
H23A	1.0390	0.2691	−0.4037	0.026*
H23B	1.1866	0.2735	−0.3935	0.026*
C24	1.12827 (14)	0.14453 (12)	−0.29159 (9)	0.0181 (3)
H24A	1.0959	0.0721	−0.2928	0.022*
H24B	1.2295	0.0951	−0.2769	0.022*
C25	1.07626 (14)	0.34487 (13)	−0.19663 (9)	0.0192 (3)
H25A	1.0905	0.3981	−0.2560	0.023*
H25B	1.1758	0.2874	−0.1802	0.023*
C26	0.96492 (15)	0.44957 (13)	−0.11458 (9)	0.0208 (3)

H26A	1.0061	0.5105	−0.1025	0.025*
H26B	0.8682	0.5123	−0.1335	0.025*
C27	0.88279 (15)	0.28513 (14)	−0.04521 (9)	0.0222 (3)
H27A	0.7851	0.3425	−0.0640	0.027*
H27B	0.8647	0.2350	0.0150	0.027*
C28	0.99553 (15)	0.17629 (13)	−0.12430 (9)	0.0197 (3)
H28A	1.0928	0.1171	−0.1052	0.024*
H28B	0.9562	0.1126	−0.1341	0.024*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0209 (5)	0.0253 (5)	0.0202 (5)	−0.0151 (4)	−0.0019 (4)	−0.0037 (4)
O2	0.0215 (5)	0.0235 (4)	0.0180 (5)	−0.0106 (4)	−0.0018 (4)	−0.0062 (4)
O3	0.0228 (5)	0.0519 (7)	0.0277 (6)	−0.0175 (5)	0.0070 (4)	−0.0198 (5)
O4	0.0196 (5)	0.0286 (5)	0.0258 (5)	−0.0094 (4)	−0.0061 (4)	−0.0070 (4)
O5	0.0196 (5)	0.0214 (4)	0.0209 (5)	−0.0100 (4)	−0.0010 (4)	−0.0069 (4)
O6	0.0290 (5)	0.0254 (5)	0.0166 (5)	−0.0158 (4)	−0.0034 (4)	−0.0028 (4)
N1	0.0177 (5)	0.0197 (5)	0.0152 (5)	−0.0093 (4)	−0.0004 (4)	−0.0020 (4)
N2	0.0167 (5)	0.0195 (5)	0.0132 (5)	−0.0089 (4)	−0.0008 (4)	−0.0013 (4)
N3	0.0181 (5)	0.0199 (5)	0.0174 (5)	−0.0061 (4)	−0.0012 (4)	−0.0033 (4)
N4	0.0173 (5)	0.0173 (5)	0.0141 (5)	−0.0087 (4)	−0.0014 (4)	−0.0028 (4)
C1	0.0158 (6)	0.0148 (5)	0.0162 (6)	−0.0071 (4)	−0.0045 (5)	0.0014 (4)
C2	0.0153 (6)	0.0170 (5)	0.0158 (6)	−0.0074 (5)	−0.0022 (5)	0.0007 (4)
C3	0.0174 (6)	0.0140 (5)	0.0151 (6)	−0.0047 (4)	−0.0048 (5)	−0.0003 (4)
C4	0.0172 (6)	0.0178 (5)	0.0216 (6)	−0.0095 (5)	−0.0074 (5)	0.0008 (5)
C5	0.0142 (5)	0.0172 (5)	0.0194 (6)	−0.0072 (5)	−0.0037 (5)	0.0027 (5)
C6	0.0144 (5)	0.0149 (5)	0.0160 (6)	−0.0060 (4)	−0.0031 (4)	0.0008 (4)
C7	0.0221 (6)	0.0224 (6)	0.0182 (6)	−0.0087 (5)	0.0009 (5)	−0.0033 (5)
C8	0.0146 (6)	0.0164 (5)	0.0180 (6)	−0.0066 (5)	−0.0022 (5)	0.0011 (4)
C9	0.0224 (6)	0.0213 (6)	0.0139 (6)	−0.0115 (5)	−0.0012 (5)	−0.0015 (5)
C10	0.0171 (6)	0.0209 (6)	0.0139 (6)	−0.0079 (5)	−0.0021 (5)	−0.0015 (5)
C11	0.0258 (7)	0.0208 (6)	0.0200 (7)	−0.0048 (5)	−0.0047 (5)	−0.0034 (5)
C12	0.0300 (7)	0.0288 (7)	0.0245 (7)	−0.0109 (6)	0.0001 (6)	−0.0090 (6)
C13	0.0389 (8)	0.0540 (9)	0.0177 (7)	−0.0346 (8)	0.0055 (6)	−0.0074 (6)
C14	0.0290 (7)	0.0415 (8)	0.0183 (7)	−0.0251 (7)	0.0019 (5)	−0.0056 (6)
C15	0.0190 (6)	0.0188 (5)	0.0159 (6)	−0.0099 (5)	−0.0034 (5)	0.0011 (4)
C16	0.0146 (6)	0.0178 (5)	0.0189 (6)	−0.0073 (5)	−0.0021 (5)	−0.0004 (5)
C17	0.0199 (6)	0.0151 (5)	0.0150 (6)	−0.0093 (5)	−0.0014 (5)	0.0001 (4)
C18	0.0187 (6)	0.0213 (6)	0.0191 (6)	−0.0110 (5)	−0.0063 (5)	0.0013 (5)
C19	0.0158 (6)	0.0184 (6)	0.0187 (6)	−0.0071 (5)	−0.0037 (5)	0.0029 (5)
C20	0.0176 (6)	0.0170 (5)	0.0152 (6)	−0.0078 (5)	−0.0026 (5)	0.0013 (4)
C21	0.0208 (6)	0.0220 (6)	0.0275 (7)	−0.0086 (5)	0.0008 (5)	−0.0084 (5)
C22	0.0176 (6)	0.0188 (6)	0.0170 (6)	−0.0080 (5)	−0.0031 (5)	0.0026 (5)
C23	0.0216 (6)	0.0195 (6)	0.0188 (6)	−0.0061 (5)	−0.0023 (5)	−0.0051 (5)
C24	0.0168 (6)	0.0170 (5)	0.0183 (6)	−0.0055 (5)	−0.0044 (5)	−0.0038 (5)
C25	0.0199 (6)	0.0207 (6)	0.0185 (6)	−0.0115 (5)	−0.0021 (5)	−0.0024 (5)
C26	0.0253 (7)	0.0204 (6)	0.0187 (6)	−0.0127 (5)	−0.0035 (5)	−0.0028 (5)

C27	0.0242 (7)	0.0271 (6)	0.0183 (6)	−0.0158 (6)	−0.0016 (5)	−0.0017 (5)
C28	0.0237 (6)	0.0206 (6)	0.0178 (6)	−0.0128 (5)	−0.0048 (5)	0.0000 (5)

Geometric parameters (Å, °)

O1—C1	1.2741 (15)	C10—H10B	0.9900
O2—C3	1.3652 (14)	C11—C12	1.5115 (18)
O2—C7	1.4342 (15)	C11—H11A	0.9900
O3—C13	1.423 (2)	C11—H11B	0.9900
O3—C12	1.4253 (18)	C12—H12A	0.9900
O4—C15	1.2673 (16)	C12—H12B	0.9900
O5—C17	1.3691 (14)	C13—C14	1.5172 (18)
O5—C21	1.4308 (15)	C13—H13A	0.9900
O6—C27	1.4274 (15)	C13—H13B	0.9900
O6—C26	1.4291 (15)	C14—H14A	0.9900
N1—C8	1.3071 (16)	C14—H14B	0.9900
N1—C9	1.4581 (16)	C15—C16	1.4436 (17)
N1—H1	0.883 (9)	C15—C20	1.4518 (17)
N2—C11	1.4657 (16)	C16—C17	1.3702 (18)
N2—C10	1.4673 (15)	C16—H16	0.9500
N2—C14	1.4676 (16)	C17—C18	1.4263 (17)
N3—C22	1.3097 (17)	C18—C19	1.3657 (17)
N3—C23	1.4625 (15)	C18—H18	0.9500
N3—H3	0.889 (9)	C19—C20	1.4187 (18)
N4—C24	1.4654 (15)	C19—H19	0.9500
N4—C28	1.4685 (16)	C20—C22	1.4068 (17)
N4—C25	1.4706 (15)	C21—H21A	0.9800
C1—C2	1.4383 (16)	C21—H21B	0.9800
C1—C6	1.4486 (16)	C21—H21C	0.9800
C2—C3	1.3724 (17)	C22—H22	0.9500
C2—H2	0.9500	C23—C24	1.5246 (18)
C3—C4	1.4229 (17)	C23—H23A	0.9900
C4—C5	1.3619 (17)	C23—H23B	0.9900
C4—H4	0.9500	C24—H24A	0.9900
C5—C6	1.4207 (17)	C24—H24B	0.9900
C5—H5	0.9500	C25—C26	1.5177 (17)
C6—C8	1.4096 (17)	C25—H25A	0.9900
C7—H7A	0.9800	C25—H25B	0.9900
C7—H7B	0.9800	C26—H26A	0.9900
C7—H7C	0.9800	C26—H26B	0.9900
C8—H8	0.9500	C27—C28	1.5138 (17)
C9—C10	1.5231 (16)	C27—H27A	0.9900
C9—H9A	0.9900	C27—H27B	0.9900
C9—H9B	0.9900	C28—H28A	0.9900
C10—H10A	0.9900	C28—H28B	0.9900
C3—O2—C7	117.10 (10)	C14—C13—H13B	109.4
C13—O3—C12	108.59 (10)	H13A—C13—H13B	108.0
C17—O5—C21	117.66 (10)	N2—C14—C13	109.57 (11)
C27—O6—C26	109.87 (9)	N2—C14—H14A	109.8

C8—N1—C9	123.55 (11)	C13—C14—H14A	109.8
C8—N1—H1	116.5 (12)	N2—C14—H14B	109.8
C9—N1—H1	119.9 (12)	C13—C14—H14B	109.8
C11—N2—C10	108.28 (10)	H14A—C14—H14B	108.2
C11—N2—C14	109.75 (10)	O4—C15—C16	122.00 (11)
C10—N2—C14	112.35 (10)	O4—C15—C20	121.65 (11)
C22—N3—C23	122.71 (11)	C16—C15—C20	116.34 (11)
C22—N3—H3	116.9 (11)	C17—C16—C15	121.19 (11)
C23—N3—H3	120.4 (11)	C17—C16—H16	119.4
C24—N4—C28	109.97 (9)	C15—C16—H16	119.4
C24—N4—C25	111.83 (9)	O5—C17—C16	124.99 (11)
C28—N4—C25	108.10 (10)	O5—C17—C18	113.13 (11)
O1—C1—C2	121.77 (11)	C16—C17—C18	121.87 (11)
O1—C1—C6	121.36 (11)	C19—C18—C17	118.51 (11)
C2—C1—C6	116.85 (11)	C19—C18—H18	120.7
C3—C2—C1	120.63 (11)	C17—C18—H18	120.7
C3—C2—H2	119.7	C18—C19—C20	122.08 (11)
C1—C2—H2	119.7	C18—C19—H19	119.0
O2—C3—C2	124.61 (11)	C20—C19—H19	119.0
O2—C3—C4	113.45 (10)	C22—C20—C19	118.70 (11)
C2—C3—C4	121.95 (11)	C22—C20—C15	121.32 (11)
C5—C4—C3	119.04 (11)	C19—C20—C15	119.98 (11)
C5—C4—H4	120.5	O5—C21—H21A	109.5
C3—C4—H4	120.5	O5—C21—H21B	109.5
C4—C5—C6	121.45 (11)	H21A—C21—H21B	109.5
C4—C5—H5	119.3	O5—C21—H21C	109.5
C6—C5—H5	119.3	H21A—C21—H21C	109.5
C8—C6—C5	119.15 (11)	H21B—C21—H21C	109.5
C8—C6—C1	120.63 (11)	N3—C22—C20	125.90 (12)
C5—C6—C1	120.06 (11)	N3—C22—H22	117.1
O2—C7—H7A	109.5	C20—C22—H22	117.1
O2—C7—H7B	109.5	N3—C23—C24	109.88 (10)
H7A—C7—H7B	109.5	N3—C23—H23A	109.7
O2—C7—H7C	109.5	C24—C23—H23A	109.7
H7A—C7—H7C	109.5	N3—C23—H23B	109.7
H7B—C7—H7C	109.5	C24—C23—H23B	109.7
N1—C8—C6	125.19 (11)	H23A—C23—H23B	108.2
N1—C8—H8	117.4	N4—C24—C23	113.23 (10)
C6—C8—H8	117.4	N4—C24—H24A	108.9
N1—C9—C10	110.56 (10)	C23—C24—H24A	108.9
N1—C9—H9A	109.5	N4—C24—H24B	108.9
C10—C9—H9A	109.5	C23—C24—H24B	108.9
N1—C9—H9B	109.5	H24A—C24—H24B	107.7
C10—C9—H9B	109.5	N4—C25—C26	110.72 (10)
H9A—C9—H9B	108.1	N4—C25—H25A	109.5
N2—C10—C9	111.97 (10)	C26—C25—H25A	109.5
N2—C10—H10A	109.2	N4—C25—H25B	109.5
C9—C10—H10A	109.2	C26—C25—H25B	109.5
N2—C10—H10B	109.2	H25A—C25—H25B	108.1

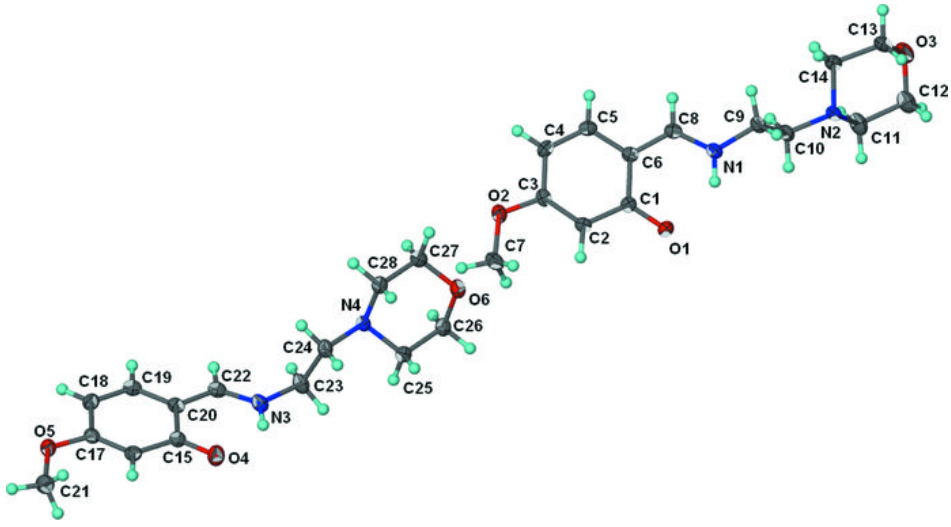
C9—C10—H10B	109.2	O6—C26—C25	111.69 (10)
H10A—C10—H10B	107.9	O6—C26—H26A	109.3
N2—C11—C12	111.64 (11)	C25—C26—H26A	109.3
N2—C11—H11A	109.3	O6—C26—H26B	109.3
C12—C11—H11A	109.3	C25—C26—H26B	109.3
N2—C11—H11B	109.3	H26A—C26—H26B	107.9
C12—C11—H11B	109.3	O6—C27—C28	111.39 (10)
H11A—C11—H11B	108.0	O6—C27—H27A	109.3
O3—C12—C11	111.69 (12)	C28—C27—H27A	109.3
O3—C12—H12A	109.3	O6—C27—H27B	109.3
C11—C12—H12A	109.3	C28—C27—H27B	109.3
O3—C12—H12B	109.3	H27A—C27—H27B	108.0
C11—C12—H12B	109.3	N4—C28—C27	109.79 (10)
H12A—C12—H12B	107.9	N4—C28—H28A	109.7
O3—C13—C14	111.10 (13)	C27—C28—H28A	109.7
O3—C13—H13A	109.4	N4—C28—H28B	109.7
C14—C13—H13A	109.4	C27—C28—H28B	109.7
O3—C13—H13B	109.4	H28A—C28—H28B	108.2
O1—C1—C2—C3	179.90 (11)	O4—C15—C16—C17	−179.19 (11)
C6—C1—C2—C3	1.07 (16)	C20—C15—C16—C17	0.26 (17)
C7—O2—C3—C2	6.74 (17)	C21—O5—C17—C16	−5.24 (18)
C7—O2—C3—C4	−172.62 (10)	C21—O5—C17—C18	175.49 (10)
C1—C2—C3—O2	−178.94 (10)	C15—C16—C17—O5	179.04 (10)
C1—C2—C3—C4	0.37 (18)	C15—C16—C17—C18	−1.75 (19)
O2—C3—C4—C5	178.02 (10)	O5—C17—C18—C19	−178.74 (10)
C2—C3—C4—C5	−1.36 (18)	C16—C17—C18—C19	1.96 (18)
C3—C4—C5—C6	0.81 (17)	C17—C18—C19—C20	−0.67 (18)
C4—C5—C6—C8	−174.64 (11)	C18—C19—C20—C22	179.23 (11)
C4—C5—C6—C1	0.66 (17)	C18—C19—C20—C15	−0.77 (18)
O1—C1—C6—C8	−5.18 (17)	O4—C15—C20—C22	0.43 (19)
C2—C1—C6—C8	173.65 (11)	C16—C15—C20—C22	−179.02 (11)
O1—C1—C6—C5	179.58 (11)	O4—C15—C20—C19	−179.57 (11)
C2—C1—C6—C5	−1.58 (16)	C16—C15—C20—C19	0.98 (17)
C9—N1—C8—C6	−172.63 (11)	C23—N3—C22—C20	−177.67 (11)
C5—C6—C8—N1	177.35 (11)	C19—C20—C22—N3	−179.06 (12)
C1—C6—C8—N1	2.07 (18)	C15—C20—C22—N3	0.94 (19)
C8—N1—C9—C10	−112.86 (13)	C22—N3—C23—C24	102.47 (14)
C11—N2—C10—C9	178.68 (11)	C28—N4—C24—C23	170.73 (10)
C14—N2—C10—C9	−59.94 (14)	C25—N4—C24—C23	−69.17 (13)
N1—C9—C10—N2	−171.02 (10)	N3—C23—C24—N4	−173.15 (10)
C10—N2—C11—C12	176.04 (11)	C24—N4—C25—C26	−178.31 (10)
C14—N2—C11—C12	53.07 (15)	C28—N4—C25—C26	−57.12 (13)
C13—O3—C12—C11	59.03 (15)	C27—O6—C26—C25	−56.66 (13)
N2—C11—C12—O3	−55.80 (16)	N4—C25—C26—O6	57.23 (14)
C12—O3—C13—C14	−61.84 (15)	C26—O6—C27—C28	58.34 (13)
C11—N2—C14—C13	−54.87 (15)	C24—N4—C28—C27	−179.29 (10)
C10—N2—C14—C13	−175.41 (11)	C25—N4—C28—C27	58.37 (13)
O3—C13—C14—N2	60.70 (16)	O6—C27—C28—N4	−60.28 (14)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.88 (1)	1.96 (1)	2.6489 (15)	133 (2)
N1—H1···O1^{i}	0.88 (1)	2.32 (1)	2.9570 (18)	129 (1)
N3—H3···O4	0.89 (1)	2.02 (1)	2.6930 (15)	132 (1)
N3—H3···O4^{ii}	0.89 (1)	2.29 (1)	2.9505 (18)	132 (1)

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+1; (ii) −*x*+3, −*y*, −*z*−1.

Fig. 1


C13
O3
C14
C12
C4
C5
C8
C9
N2
C11
C10
C6
N1
O2
C3
C1
C2
O1
C7
C27
C28
O6
N4
C26
C24
C23
C25
C18
C19
C22
C20
N3
O5
C17
C15
O4
C21